

Se. 09/684,433



PATENT OFFICE
JAPANESE GOVERNMENT

This is to certify that the annexed is a true copy of the following application as filed with this Office.

Date of Application: April 27, 2000

Application Number: Patent Application
2000-127082

Applicant(s): Shimadzu Corporation

August 11, 2000

Commissioner,
Patent Office Kozoh Oikawa (sealed)

Certificate No. 2000-3063820

[Document Name] Patent Application

[Document Number] K1000195

[Date of Submission] April 27, 2000

[Addressee] Commissioner, Patent Office

[Classification of International Patent] A23L 3/00

[Title of the Invention] CONTINUOUS PROCESS METHOD AND CONTINUOUS PROCESS APPARATUS FOR LIQUID-FORM SUBSTANCE, AND LIQUID-FORM SUBSTANCE PROCESSED THEREBY

[Number of Claims] 3

[Inventor]
[Address or Dwelling Place]
[Name] 20-17 Mikkaichimachi, Shobara-shi,
Hiroshima-ken, Japan

[Name] Yutaka Osajima

[Inventor]
[Address or Dwelling Place]
[Name] 1-19-18 Mainosato, Koga-cho, Kasuya-gun,
Fukuoka-ken
[Name] Mitsuya Shimoda

[Inventor]
[Address or Dwelling Place]
[Name] c/o Shimadzu Corporation
1, Nishinokyo-kuwabaracho, Nakagyo-ku,
Kyoto
[Name] Masaki Miyake

[Patent Applicant]
[ID No.] 000001993
[Name or Title] Shimadzu Corporation

[Agent]
[ID No.] 100095670
[Patent Attorney]
[Name or Title] Ryohei Kobayashi

[Indication of Fee]
[Prepayment Note No.] 019079
[Amount of Payment] 21,000 yen

[List of Submission]
[Name] Specification 1
[Name] Drawing 1
[Name] Statement of Abstract 1

Comprehensive Power of Attorney No. 9116525

[Proof to be required or not] Required

[Document Name] Specification

[Title of the Invention]

CONTINUOUS PROCESS METHOD AND CONTINUOUS PROCESS APPARATUS FOR LIQUID-FORM SUBSTANCE, AND LIQUID-FORM SUBSTANCE PROCESSED THEREBY

[Scope of Patent Claim]

[Claim 1] A continuous process method of a liquid-form substance for continuously processing a liquid-form raw material, such as a liquid-form food, by using supercritical fluid or subcritical fluid, characterized by comprising:

a solution step for continuously supplying a liquid carbon dioxide into a continuously supplied liquid-form raw material to thereby dissolve the liquid carbon dioxide in the liquid-form raw material;

a holding step for holding the liquid-form raw material, for a predetermined time, into which the liquid carbon dioxide has been dissolved at the solution step;

a critical process step for holding the liquid-form raw material into which the liquid carbon dioxide has been dissolved under predetermined temperature and pressure conditions to thereby change the carbon dioxide to a supercritical state or a subcritical state; and

a decompressing step for suddenly decompressing the liquid-form raw material having passed through the critical process step to thereby remove the carbon dioxide and, at the same time, collect a product.

[Claim 2] A continuous process apparatus for continuously processing a liquid-form raw material by using a supercritical fluid or a subcritical fluid, characterized by comprising:

- a) a raw material supply path for continuously supplying a liquid-form raw material;
- b) a carbon dioxide supply path for continuously supplying a liquefied carbon dioxide;
- c) a solution portion for dissolving the liquid carbon dioxide supplied through the carbon dioxide supply path into the liquid-form raw material supplied through the raw material supply path;
- d) a holding portion for holding, for a predetermined time, the liquid-form raw material into which the liquid carbon dioxide has been dissolved at the solution step;
- e) a critical process portion for taking out the liquid-form raw material, into which the liquid carbon dioxide has been dissolved, from the solution portion and holding thereof under predetermined temperature and pressure conditions to thereby change the carbon dioxide to a supercritical state or a subcritical state; and
- f) a decompression portion for suddenly decompressing the liquid-form raw material having passed through the critical process portion so that the carbon dioxide is removed and, at the same time, a product is collected.

[Claim 3] A liquid-form substance processed and collected by the continuous process method as claimed in claim 1 or the continuous process apparatus as claimed in claim 2.

[Detailed Description of the Invention]

[0001]

[Technical Field of the Invention]

This invention relates to a continuous process method and a continuous process apparatus for continuously carrying out deactivation and sterilization processes of enzymes and spores of liquid-form foods and liquid-form medical supplies or deodorization process of the liquid-form foods or the like by using a supercritical or sub-critical fluid, and a liquid substance (for example, liquid-form food or drink and liquid-form medical supply) obtained by the above method and apparatus.

[0002]

[Prior Art]

There are various kinds of liquid-form foods containing enzymes, typically such as a refined sake, beer and fruit juice. A manufacturing process of the refined sake includes a first step wherein a young sake can be obtained by carrying out compression/filtration after completion of a fermentation; a second step wherein the obtained young sake is heated and sterilized to be stored; a third step wherein the obtained original sake is mixed to determine its quality and, at the same time, to allow its alcoholic content to match the standard; and a fourth step wherein the adjusted sake is again heated to sterilize

and filled in a bottle or a paper pack. As described above, in the refined sake which has been subjected to two times of heating processes, the enzymes are deactivated and sterilized so that the quality of the refined sake during its distribution can be prevented from being deteriorated. However, such a heating process extremely reduces a fresh flavor and taste of the young sake. Therefore, in order to enjoy such fresh flavor and taste, there has been produced a green sake wherein the heating process is not carried out. The green sake is distributed at a cold temperature in order to protect its quality. However, such a green sake which has not been subjected to the heating process is liable to deteriorate in its quality due to actions of enzymes, such as an α -amylase, protease or the like. Further, there is a problem that its cost is raised because of the cold temperature distribution.

[0003]

Also, in order to hold a stability of a turbid juice, such as an orange juice, an inactivation of pectinesterase (PE) is required. Since the PE is a stable enzyme against heat, in order to carry out inactivation of the PE by heating, a heat process under the condition of a high temperature (88 to 99 °C or 120°C) is required. However, there is a problem such that when the heat process under such a high temperature condition is carried out, the flavor of the juice may be deteriorated.

[0004]

In view of the problems as described above, the present inventors have already proposed a novel technique wherein an

enzyme containing liquid-form food is subjected to contact with a supercritical state carbon dioxide to thereby deactivate the enzymes (refer to Japanese Patent Publication "TOKKAIHEI" 7-170965). In the technique, an enzyme containing liquid-form food is stored in a process tank and sealed, the interior of the sealed tank is held under the condition of a predetermined temperature and pressure, and, at the same time, a supercritical fluid of carbon dioxide is supplied into the process tank through a filter to be a fine size (an average diameter of less than several hundreds μm), so that the supercritical fluid can be easily dissolved in the liquid-form food. According to the method, the enzyme can be effectively deactivated and, at the same time, since only the carbon dioxide contacts the food, there is an advantage that a high safety can be obtained. Also, according to the method, microorganisms, such as bacteria, yeast and mold, can be sterilized at the same time.

[0005]

Further, the present inventors have proposed a continuous process apparatus for carrying out the deactivation process/sterilization process effectively and without deterioration of quality of products (Refer to Japanese Patent Publication "TOKKAIHEI" 9-206044, U.S. Patent No.5,704,276). In the continuous process apparatus, a liquid-form food is continuously supplied to a bottom portion of the process tank held at a predetermined pressure and temperature; at the same time, a supercritical state carbon dioxide is continuously supplied to the bottom portion of the process tank through a mesh-type filter

disposed thereat; and a liquid outlet is provided at an upper portion of the process tank in the vicinity of an under-liquid-surface to thereby collect a product. The liquid-form food and the fine bubble-form supercritical fluid contact each other while making a parallel flow in an elevating direction in the process tank to thereby effectively deactivate the enzymes. Also, a supercritical fluid discharge port is disposed at an upper portion of the process tank, through which the supercritical fluid is taken out and returned to a carbon dioxide supply source to reuse. According to the apparatus, since the liquid-form food can be continuously processed, the apparatus is suitable for a factory which processes a large amount of drinks or foods.

[0006]

[Problems to be Solved by the Invention]

According to the above explained continuous process apparatus, the deactivation/sterilization process of enzymes can be continuously carried out at a high efficiency. However, in case the continuous process apparatus is put to practical use, there is a problem, especially, in its cost. More specifically, in the above continuous process apparatus, it is required that the process tank is held at a temperature higher than 31.1°C in order to hold carbon dioxide in a supercritical state. However, a solubility of carbon dioxide into the liquid-form food becomes lower as the temperature becomes higher. Thus, in view of solution, its efficiency is poor. Therefore, in order to obtain sufficient deactivation and sterilization effects, it is required that the liquid-form food and the supercritical fluid are

subjected to a parallel flow for a predetermined time (in the order of several to several tens of minutes). Therefore, it is necessary to shorten time by making the process tank in a large capacity. Also, in order to hold the process tank at the above-stated temperature, it is required to provide a heater. Further, since a reaction in the process tank becomes slow when a temperature of the liquid-form food to be supplied to the process tank is low, a heater for properly heating the liquid-form food is required before the food is supplied to the process tank. As described above, the continuous process apparatus requires an equipment on a large scale, which results in a large occupancy area as well as a high cost.

[0007]

Also, the temperature in the process tank is considerably lower than that for deactivating enzymes by heating, but higher than the room temperature. Thus, in case the liquid-form food is left as it is under such a temperature condition for the predetermined time, the quality of the liquid-form food may be deteriorated. More specifically, for example, a citrus juice right after squeezed contains high active enzymes, so that the enzymes may act on the juice in the process tank before the enzymes are deactivated to deteriorate a quality of the juice.

[0008]

To solve these problems, the present invention has been made, and a main object thereof is to provide a continuous process method, a continuous process apparatus and a liquid-form substance

processed thereby, wherein a process tank can be miniaturized and the number of heaters to be disposed can be minimized.

[0009]

[Means for Solving the Problems]

In the continuous process apparatus disclosed in the previous application, a process for dissolving carbon dioxide in a liquid-form food and a process for allowing the carbon dioxide to be a supercritical state and holding in its state, are carried out in a process tank simultaneously. On the contrary, in a continuous process method and a continuous process apparatus according to the present invention made to solve the above problems, it is characterized in that the two processes disclosed in the previous applications are carried out separately timewise and spacewise.

[0010]

In other words, the continuous process method according to the present invention, in which a liquid-form raw material, such as a liquid-form food, is continuously subjected to a process by using a supercritical or subcritical fluid, is characterized by including:

a solution step for continuously supplying a liquid carbon dioxide to a continuously supplied liquid-form raw material to thereby dissolve the liquid carbon dioxide in the liquid-form raw material;

a holding step for holding the liquid-form raw material, for a predetermined time, into which the liquid carbon dioxide has been dissolved at the solution process;

a critical process step for holding the liquid-form raw material into which the liquid carbon dioxide has been dissolved under predetermined temperature and pressure conditions to thereby allow the carbon dioxide to be a supercritical or sub-critical state; and

a decompressing step for suddenly decompressing the liquid-form raw material having passed through the critical process step to thereby remove the carbon dioxide and collect a product.

[0011]

Also, a continuous process apparatus of the present invention embodying the above continuous process method, in which a liquid-form raw material, such as a liquid-form food, is continuously processed by using a supercritical or subcritical fluid, is characterized by including:

- a) a raw material supply path for continuously supplying a liquid-form raw material;
- b) a carbon dioxide supply path for continuously supplying a liquefied carbon dioxide;
- c) a solution portion for dissolving the liquid carbon dioxide supplied through the carbon dioxide supply path in the liquid-form raw material supplied through the raw material supply path;
- d) a holding portion for holding, for a predetermined time, the liquid-form raw material into which the liquid carbon dioxide has been dissolved at the solution step;
- e) a critical process portion for taking out the liquid-form raw material, into which the liquid carbon dioxide has been

dissolved, from the solution portion and holding thereof under predetermined temperature and pressure conditions to thereby allow the carbon dioxide to be a supercritical or subcritical state; and

f) a decompressing portion for suddenly decompressing the liquid-form raw material having passed through the critical process portion to remove the carbon dioxide and collecting a product.

[0012]

Further, a liquid-form substance according to the present invention is characterized by a liquid-form substance processed and collected by the above-stated continuous process method or continuous process apparatus.

[0013]

[Embodiments of the Invention]

In the continuous process method and continuous process apparatus according to the present invention, while a liquid-form raw material, such as a liquid-form food and liquid-form chemical, is continuously supplied to the solution portion through a raw material supply path, a cooled and liquefied carbon dioxide, (hereinafter referred to "liquid carbon dioxide") is continuously supplied to the solution portion through the carbon dioxide supply path. A mesh-type filter having, for example, fine diameter holes is disposed at an outlet of the carbon dioxide supply path, and when passing through the filter, the liquid carbon dioxide becomes fine bubbles to thereby dissolve into the liquid-form raw material. Of course, a contact efficiency of the carbon dioxide and the liquid-form raw material may be increased by the other method,

such as a high speed mixer and an ultrasonic wave generator. As is generally known, a solubility of the liquid carbon dioxide into a liquid is higher as the circumferential temperature is lower. Therefore, while it is desirable that the solution portion is cooled, even if the temperature thereof is the room temperature, a sufficient quantity of the liquid carbon dioxide can be dissolved in the liquid-form raw material for a short time. Especially, in a winter season, since the ambient temperature is low, the solubility efficiency is high.

[0014]

For example, the solution portion includes a solution tank, and an inlet of the liquid-form raw material from the raw material supply path and an inlet of the liquid carbon dioxide from the carbon dioxide supply path are disposed at a bottom portion of the solution tank, and an outlet for taking out the liquid can be located in the vicinity of a liquid surface at an upper portion of the solution tank. According to the structure, the liquid-form raw material introduced through the bottom portion of the solution tank flows in the solution tank to elevate therethrough, and the bubble-shape liquid carbon dioxide also flows in the same direction. Therefore, contact areas thereof are extremely wide, so that the liquid carbon dioxide can be effectively dissolved in the liquid-form raw material.

[0015]

Also, the above solution portion may be structured such that the liquid carbon dioxide is dissolved in the liquid-form raw material by supplying the liquid carbon dioxide into the liquid-

form raw material flowing through a raw material supply pipe used as a raw material supply path. As described above, in case the carbon dioxide is dissolved in the liquid-form raw material in the raw material supply pipe, it is not required to provide a special tank for dissolving the carbon dioxide on the way of the raw material supply pipe to thereby miniaturize the entire apparatus.

[0016]

As a method for effectively dissolving the liquid carbon dioxide into the liquid-form raw material flowing in a raw material supply pipe, there are, for example, a method wherein a mesh-type filter is disposed in the raw material supply pipe to allow the liquid carbon dioxide to pass therethrough, so that the fine bubbles of the liquid carbon dioxide are released in the liquid-form raw material; and a method wherein a mixer for mixing liquids is provided on the way of the raw material supply pipe, and the liquid carbon dioxide is supplied to the liquid-form raw material on an upstream side than the mixer. Incidentally, as stated above, since the solubility of the liquid carbon dioxide into a liquid is higher as an ambient temperature is lower, it is preferable to cool the raw material supply pipe at portions where the filter and mixer are provided. However, at this time, it is not always necessary to cool the raw material supply pipe to a specially low temperature. For example, even if it is the room temperature, a sufficient amount of the liquid carbon dioxide can be dissolved in the liquid-form raw material for a short time. Especially, in a winter season, since the ambient temperature is low, the dissolving efficiency is high. Therefore, for example,

it is effective only to provide a device for keeping the above-stated portions of the raw material supply pipe warm.

[0017]

The liquid-form raw material into which the liquid carbon dioxide has been dissolved at the solution portion is sent to the holding portion. The holding portion is structured such that the liquid carbon dioxide can act on substances to be processed (enzymes, microorganisms or the like) in the liquid-form raw material for a sufficiently long time. A temperature of the holding portion is set at a temperature lower than that of the critical process portion, described later (for example, substantially the same temperature as that of the solution portion). As a specific shape of the holding portion, for example, there are mentioned a spiral pipe; a tank having a sufficient capacity with respect to a flow quantity of the liquid-form raw material; a tank provided with a baffle therein; a tank provided with a spiral screw structure on an inner wall surface; and a tank provided with a screw-type structure therein. Or, a tank is divided into two chambers by a partition or like, one chamber on the upstream side may constitute the solution portion and the other chamber on the downstream side may constitute the holding portion. In case the holding portion is provided, since the liquid carbon dioxide is sufficiently penetrated into protein for constituting objects to be processed (enzymes, microorganisms and the like), for example, even if strong bacteria can be positively killed in the critical process step and decompression step, described later.

[0018]

Incidentally, as a flow path for sending the liquid-form raw material to the critical process portion, described later, from the solution portion, in addition to a first flow path passing through the holding portion, a second flow path which does not pass through the holding portion may be provided separately, so that either one of the flow paths may be selected according to a kind of the process or a kind of a substance to be processed. Further, a holding portion may be formed of a plurality of independently operable holding units, and one, a part or all of the holding units may be selected as an operable unit as an occasion arises.

[0019]

The liquid-form raw material into which the liquid carbon dioxide has been dissolved at the solution portion is sent to the next critical process portion. The critical process portion is held under the condition of a temperature and pressure required for converting the carbon dioxide in a supercritical or subcritical state. As such a condition, it is preferable to hold a temperature of 30 to 80 °C, preferably 30 to 50°C and a pressure of 40 to 400 atm, preferably 100 to 300 atm. Under the condition, the liquid carbon dioxide dissolved in the liquid-form raw material is suddenly changed to the supercritical or subcritical state. It is sufficient that a staying time of the liquid-form raw material in the heating tank is at the most in the order of one minute. Therefore, although the temperature is higher than

the room temperature, deterioration of a quality of the liquid-form raw material can be suppressed to the minimum limit.

[0020]

In the decompression step carried out at the next decompression portion, the pressure of the liquid-form raw material subjected to the above process at the critical process portion is suddenly reduced. Then, the carbon dioxide having penetrated into the protein as an active substance of the enzymes is suddenly expanded and the protein is destructed to deactivate the enzymes. Also, sterilization of various microorganisms can be carried out. Since the carbon dioxide dissolved in the liquid-form raw material is vaporized and volatilized from the liquid-form raw material, the liquid-form raw material can be collected as a product after the process. In such a decompression process, a decompression speed is important. For example, in case the decompression is carried out by using a pressure controlling valve with an orifice, it is preferable to set the decompression speed such that the liquid-form raw material passes through the orifice at a speed of less than 20 mm/sec, preferably less than 10 mm/sec.

[0021]

Incidentally, as the liquid-form raw material to which the present invention is applied, a fermentation/brewing liquid-form foods, such as raw sake, beer, wine and soy source, various fruit juices and soft drinks are typically mentioned. Although the fruits juices are generally produced from apples, grapes and various citruses as a raw material, squeezed liquids produced from tomatoes and other vegetables as a raw material may be included.

Also, the liquid-form raw material may not be foods, and it may be a liquid-form chemical, such as various infusions, blood formulations and nutrition supply liquids.

[0022]

[Effects of the Invention]

As described hereinabove, according to the continuous process method and continuous process apparatus of a liquid-form substance of the present invention, since the solution step of the liquid carbon dioxide into the liquid-form raw material and the critical process step for changing the carbon dioxide into a supercritical or subcritical state are separated, the respective steps can be extremely effectively carried out, so that the overall process time can be greatly shortened when compared with those of the conventional continuous process methods and apparatuses. Therefore, a large process tank and a heater for heating the liquid-form raw material are not required, so that the apparatus can be miniaturized. Also, since the temperature setting at the critical process step can be optimized, higher effects of deactivation/sterilization of the enzymes can be obtained when compared with those in the prior arts. Further, since the time for which the liquid-form raw material is held in a warmed state is short, the flavor of the product is rarely deteriorated.

[0023]

[Embodiments]

Hereunder, embodiments of a continuous process apparatus according to the present invention will be explained with reference to the accompanying drawings.

[0024]

Fig. 1 is a constitutional diagram showing a continuous enzyme deactivation process apparatus. In the apparatus, a liquid-form raw material is stored in a raw material tank 1, and a bottom of the raw material tank 1 and a bottom of a solution tank 11 are connected by a raw material supply path 3. In the middle of the flow path 3, there is provided a pump 2 for sending the liquid while pressurizing, and the liquid-form raw material is continuously supplied to the solution tank 11 at a desired flow rate by suitably setting operating conditions of the pump 2.

[0025]

On the other hand, between a liquid carbon dioxide bomb 4 and the bottom of the solution tank 11, there is extended a carbon dioxide supply path 10 provided with a valve 5, line filter 7, cooler 8 and pump 9. The cooler 8 is provided to cool and liquefy the gaseous carbon dioxide in case the carbon dioxide is vaporized in the middle of the supply path, or the gaseous carbon dioxide supplied through a recycling path 30, described later, and the carbon dioxide held in a liquid state is pressurized by the pump 9 to be supplied to the solution tank 11.

[0026]

The solution tank 11 is formed of a withstand pressure vessel, and an outlet of the raw material supply path 3 disposed to the bottom portion thereof is provided with an introduction port 12 and an outlet of the carbon dioxide supply path 10 is provided with a mesh-type filter 13 having fine holes. In order to effectively dissolve the liquid carbon dioxide in the liquid-form

raw material, it is preferable to release the liquid carbon dioxide as the finest particles possible in the raw material. Thus, it is preferable that a mesh of the filter 13 is in a range of less than 100 μm , preferably less than 20 μm . A drain 14 for waste liquid, which can be freely closed and opened by a valve, is connected to the bottom of the solution tank 11. On the other hand, a liquid outlet 18 is disposed in the vicinity of a liquid surface on an upper portion in the solution tank 11. In other words, the liquid-form raw material introduced through the introduction port 12 flows upward in the solution tank 11, and when the liquid-form raw material reaches in the vicinity of the liquid surface, the raw material is taken out through the liquid outlet 18.

[0027]

A liquid level sensor 16 is provided in the solution tank 11, and the liquid level in the solution tank 11 is held substantially constant by feeding back an output signal of the liquid level sensor 16 to the pump 2 to thereby control its operation. Incidentally, instead of the liquid level sensor 16, for example, there may be employed controlling means for equalizing a liquid quantity taken out from the liquid outlet 18 with a liquid quantity to be supplied from the introduction port 12 so as to hold the liquid level in the solution tank 11 constant. As described above, by holding the liquid level constant, since a time for the liquid-form raw material to pass through the solution tank 11 can be maintained constant, the carbon dioxide can be

stably and uniformly dissolved in the liquid-form raw material, as described later.

[0028]

A carbon dioxide discharge port 17 is disposed to a lid portion for closing an upper end surface of the solution tank 11. While the liquid carbon dioxide is dissolved in the liquid-form raw material in the solution tank 11 as described later, depending on conditions, a part of the liquid carbon dioxide may change to a subcritical state or a supercritical state. In that case, since a density of the subcritical or supercritical fluid is less than a liquid density, the carbon dioxide in the subcritical state or the supercritical state can be taken out through the carbon dioxide discharge port 17 positioned at a higher level than the liquid surface.

[0029]

A spiral-shape heating wiring 20 is connected to the liquid outlet 18. The heating wiring 20 is held in a warming tank (or a warming metal block or the like) provided with a heater 19, and a temperature of the heating wiring 20 can be monitored by a temperature sensor 21. The temperature monitored by the temperature sensor 21 is fed back to the heater 19 to thereby keep the temperature of the heating wiring 20 substantially constant.

[0030]

On the other hand, a pressure in the solution tank 11 is monitored by a pressure sensor 15. Since the solution tank 11 and the heating wiring 20 are disposed between the pumps 2, 9 and the pressure controlling valves 22, 28, the pressure in the solution

tank 11 can be controlled to a predetermined pressure value by supplying speeds of the liquid-form raw material and the liquid carbon dioxide by the pumps 2, 9 and opening degrees of the pressure controlling valves 22, 28.

[0031]

A product collecting path 23 having the pressure controlling valve 22 for attaining a quick decompression on its way is connected to an outlet of the heating wiring 20, and the terminal edge thereof is connected to a decompression tank 24. In the decompression tank 24, the carbon dioxide having dissolved in a product (processed liquid-form raw material) is vaporized to take out, and the gaseous carbon dioxide can be returned to a recycling path 30 through a valve 25. The product collected in the decompression tank 24 is transferred to a product tank 26. Incidentally, since vaporization heat is removed when the carbon dioxide is vaporized in the decompression tank 24, the liquid-form raw material warmed in the heating wiring 20 is cooled in the decompression tank 24 to thereby obtain a product of a room temperature or lower temperature.

[0032]

A carbon dioxide collecting path 27 to be connected to a recycling tank 29 through the pressure controlling valve 28 is connected to the carbon dioxide discharge port 17. The supercritical or subcritical fluid sent to the carbon dioxide collecting path 27 is decompressed at the pressure controlling valve 28 to be a gaseous carbon dioxide and collected into the recycling tank 29. The recycling path 30 to be connected to a

carbon dioxide supplying path 10 is connected to the recycling tank 29 through a check valve 31 and valve 6 and functions as another carbon dioxide supplying source. In other words, only an insufficient portion of a carbon dioxide quantity recycled through the recycling path 30 is supplied from the liquid carbon dioxide bomb 4 to thereby save a quantity of the liquid carbon dioxide supplied therefrom.

[0033]

Next, the deactivation process of enzymes in the above apparatus is explained. The liquid-form raw material is continuously introduced into the solution tank 11 through the introduction port 12. When the liquid carbon dioxide sent through the carbon dioxide supply path 10 passes through the filter 13, the carbon dioxide is released into the liquid-form raw material as fine bubbles according to a diameter of holes of the filter 13. In other words, the liquid introduced in a form of fine bubbles through the filter 13 and the liquid-form raw material right after introduction immediately contact each other, so that the liquid carbon dioxide is effectively dissolved into the liquid-form raw material. Since such solution can be accelerated as an ambient temperature is lowered, it is preferable to cool the solution tank 11. However, by only keeping the temperature around the solution tank 11 at a room temperature, a sufficiently high solubility can be obtained.

[0034]

The liquid-form raw material, in which the liquid carbon dioxide has been dissolved, rises in the solution tank 11 and

reaches the liquid outlet 18. In order to obtain sufficient enzyme deactivation and sterilization effects, it is preferable to raise a solubility of the carbon dioxide with respect to the liquid-form raw material as much as possible. Also, in case bacteria having a strong life force intends to be killed, it is important to take a sufficient time for allowing the liquid carbon dioxide to act on the bacteria in the liquid-form raw material. A structure wherein an acting time of the liquid carbon dioxide can be prolonged is explained later.

[0035]

The liquid-form raw material taken out from the liquid outlet 18 is introduced into the heating wiring 20. At this time, although a liquid carbon dioxide, which can not be dissolved in the liquid-form raw material, (i.e. in a mixed state), is also introduced into the heating wiring 20, nothing is wrong. The heating wiring 20 is held at a temperature in the order of 30 to 40°C by the heater 19. Also, the pressure in the heating wiring 20 (and also in the solution tank 11) is held at 100 to 300 atm. Under such temperature and pressure conditions, the liquid carbon dioxide is changed to a supercritical fluid in a short time. The liquid-form raw material passes through the spiral heating wiring 20 for about one minute. When the liquid carbon dioxide having dissolved in the liquid-form raw material is transformed into the supercritical fluid, a portion of protein which is an active substance of the enzymes contained in the liquid-form raw material is destructed to thereby cause a portion of the microorganisms to

die out. However, at this point, the effect thereof can be found only in a portion.

[0036]

Then, when the liquid-form raw material reaches the decompression tank 24 through the pressure controlling valve 22, the pressure is suddenly lowered to thereby release the supercritical state and the carbon dioxide is vaporized at a stroke to suddenly be subjected to a cubical expansion. At that time, the protein of the enzymes is destructed to thereby cause the microorganisms to die out. Thus, the deactivation of the enzymes and spores and sterilization of the microorganisms can be carried out, and the processed product is collected into the product tank 26. As a result, the product collected in the product tank 26 has an extremely low active enzyme ratio so that undesired microorganisms become zero. Also, as described before, the temperature of the product at the time of collection is low. Also, since heat is not applied when the carbon dioxide is vaporized at the decompression tank 24, a flavor component itself contained in the liquid-form raw material scarcely volatilizes, so that the flavor is not spoiled or lost.

[0037]

Next, enzyme deactivation effects obtained through an experiment using the continuous enzyme deactivation process apparatus as shown in Fig. 1 are explained. Fig. 2 shows results obtained by examining ratios of the active enzymes remaining in the liquid-form raw material processed by the apparatus as shown in Fig. 1. As Comparative Examples, there are shown process

results obtained by using a continuous process apparatus (the apparatus disclosed in Japanese Patent Publication TOKKAIHEI 9-206044 as described before) wherein a conventional subcritical/supercritical fluid is used (as Comparative Example 1); and process results obtained by a heating process method which is generally used at present (as Comparative Example 2).

Here, the liquid-form raw material was supplied at a ratio of 20 Kg/h, and carbon dioxide was supplied at a ratio of 1.6 kg/h. In the Present Example, a critical process step was carried out for one minute at a temperature of 50°C under a pressure of 250 atm. On the other hand, at Comparative Example 1, a critical process step was carried out for 15 minutes at a temperature of 40°C under a pressure of 250 atm; and at Comparative Example 2, a heating process was carried out for one minute at a temperature of 85°C.

[0038]

As apparent from Fig. 2, two methods using the subcritical/supercritical fluid, i.e. the Present Example and Comparative Example 1, show higher enzyme deactivation effects than that of the heating process method of Comparative Example 2. Also, there is no significant difference between the deactivation effects of the Present Example and Comparative Example 1. In other words, by the process method according to the Present Example wherein the critical process step was carried out in a very short time, such as only one minute, there can be obtained the same deactivation effect as that of Comparative Example 1 wherein the critical process step requires 15 minutes.

[0039]

At the same time as the above experiment, the remaining spore number of ascospore microbe was also examined, and it was confirmed that remaining spore numbers of *Bacillus subtilis* and eight other bacteria of *Bacillus* were zero. In other words, by the process method according to the present invention, microorganisms can be completely sterilized.

[0040]

Also, in case there is structured an apparatus for processing a liquid-form raw material (such as fruit juices) wherein its flavor component is desired to be held as much as possible, there may be provided a path for returning at least a part of the carbon dioxide in the supercritical state taken out from the carbon dioxide discharge port 17 to the decompression tank 24, separately. When the carbon dioxide in the supercritical state flowed into the decompression tank 24 through the path is vaporized therein, the flavor component taken in the carbon dioxide in the solution tank 11 is released. The flavor component is dissolved again in the product collected in the decompression tank 24. Thus, the collected product contains much more flavor component.

[0041]

Fig. 3 is a block diagram showing another continuous enzyme deactivation process apparatus, and Fig. 4 is an enlarged view of a portion surrounded by a rectangle R in Fig. 3.

[0042]

In the apparatus shown in Fig. 3, a liquid-form raw material to be processed is stored in a raw material tank 51 having a

liquid outlet 51a. The liquid outlet 51a of the raw material tank 51 is connected to a liquid inlet 54a of a process unit 54 through a raw material supply pipe 53 on the way of which a pump 52 is disposed. On the other hand, liquid carbon dioxide to be used for processing is stored in a bomb 55. One end (inlet end) of a carbon dioxide supply pipe 60, on the way of which a valve 56, line filter 57, cooler 58 and pump 59 are disposed, is connected to a gas outlet 55a of the bomb 55. The other end (outlet end) of the carbon dioxide supply pipe 60 is inserted into the raw material supply pipe 53 to penetrate through a side wall thereof on the way of the raw material supply pipe 53. A mesh-type filter 61 having fine holes is provided in the vicinity of the outlet end of the carbon dioxide supply pipe 60. Also, the raw material supply pipe 53 is provided with a pressure sensor 53a for detecting a pressure therein in the vicinity of the liquid inlet 54a of the process unit 54.

[0043]

The process unit 54 is a warming tank including a heater 54b and a temperature sensor 54c, and has a spiral heating wiring 62 therein. One end of the heating wiring 62 is connected to the liquid inlet 54a and the other end thereof is connected to a liquid outlet 54d of the process unit 54 therein. The liquid outlet 54d of the process unit 54 is connected to one end of a product collecting path 64 having a pressure controlling valve 63. The other end of the product collecting path 64 is connected into a liquid inlet 65b provided to a side wall of the decompression tank 65 having a pressure sensor 65a. A product outlet 65c is

provided to a bottom portion of the decompression tank 65, and right under thereof a product tank 66 is located. A carbon dioxide discharge port 65d is located on an upper portion of the decompression tank 65, and one end of a carbon dioxide collecting path 68 provided with a pressure controlling valve 67 on the way is connected to the carbon dioxide discharge port 65d. The other end of the carbon dioxide collecting path 68 is connected to a gas inlet 69a provided to an upper portion of a recycling tank 69. A gas outlet 69b is provided to a bottom portion of the recycling tank 69, to which one end of a recycling pipe 70 is connected. A check valve 71 and a valve 72 are disposed on the way of the recycling pipe 70. The other end of the recycling pipe 70 is connected to the carbon dioxide supply pipe 60 between the valve 56 and the line filter 57.

[0044]

Also, not shown, the above-described apparatus includes a controlling device for driving various portions of the apparatus based on output signals of the pressure sensor and the temperature sensor. For example, the controlling device functions as a temperature controlling device for holding a temperature in the process unit 54 at a predetermined value by feed-back controlling the heater 54b based on an output signal of the temperature sensor 54c. Also, the controlling device functions as a pressure controlling device for holding pressures in the heating wiring 62 and the decompression tank 65 at predetermined values, respectively, by feed-back controlling the pumps 52, 59 and the pressure controlling valves 63, 67 based on output signals of the

pressure sensors 53a, 65a. In addition to the above, the controlling device further keeps a pressure in the raw material supply pipe 53 (which is also a pressure in the heating wiring 62) to be detected by the pressure sensor 53a, in the order of 100 to 300 atm and, at the same time, keeps a pressure in the decompression tank 65 to be detected by the pressure sensor 65a at a value (about 2 - 40 atm) further lower than that.

[0045]

The operations of the above-described apparatus are as follows. First, when the apparatus is started, the valve 56 disposed on the carbon dioxide supply pipe 3 is opened to start the pumps 52, 59. When the pump 52 starts, the liquid-form raw material stored in the raw material tank 51 is continuously supplied to the raw material supply pipe 53. Also, when the pump 59 starts, the liquid carbon dioxide stored in the bomb 55 is fed into the raw material supply pipe 53 through the carbon dioxide supply pipe 60. Here, for example, even if the liquid carbon dioxide flowed out from the bomb 55 is vaporized in the carbon dioxide supply pipe 60 to be gas, since the carbon dioxide gas is again liquefied by the cooler 58, the liquid carbon dioxide is stably supplied to the raw material supply pipe 53.

[0046]

The liquid carbon dioxide flowing through the carbon dioxide supply pipe 60 passes through the filter 61 when it flows out from the outlet end of the carbon dioxide supply pipe 60 to thereby be fine bubbles and dispersed in the liquid-form raw material (Refer to Fig. 4). Thus, since the fine bubble-form liquid introduced

through the filter 61 and the liquid-form raw material right after introduction are immediately contacted each other, the liquid carbon dioxide can be effectively dissolved in the liquid-form raw material. Incidentally, in order to effectively dissolve the liquid carbon dioxide into the liquid-form raw material, it is preferable to release the liquid carbon dioxide into the liquid-form raw material to be as fine particles as possible. Thus, the mesh of the filter 61 is less than 100 μm , more preferably less than 20 μm . Also, generally, since a gas is more dissolved in a liquid the lower a temperature is, it is preferable to cool at least a portion of the raw material supply pipe 53 where the filter 61 is provided therein. However, instead of specially cooling, for example, when the peripheral portion of the above-mentioned portion is merely kept at a room temperature, a sufficiently high solubility can be obtained.

[0047]

Incidentally, in Fig. 4, although only one filter 61 is disposed in the raw material supply pipe 53, a plurality of filters 61 may be provided. Such examples are shown in Figs. 5. More specifically, in the example of Fig. 5(a), two filters 61 are disposed in the raw material supply pipe 53 to face each other. Also, in the example of Fig. 5(b), four filters 61 are disposed alternately along a flowing direction of the liquid-form raw material. With such an arrangement, a dissolving efficiency of the liquid carbon dioxide can be further elevated. Incidentally, pipes for feeding carbon dioxide to the respective filters 61 may

be structured by, for example, diverging the carbon dioxide supply pipe 60 on the downstream side than the pump 59.

[0048]

As described above, the liquid-form raw material into which the liquid carbon dioxide has been dissolved is introduced into the heating wiring 62 through the liquid inlet 54a of the process unit 54. At this time, although the liquid carbon dioxide which can not be dissolved in the liquid-form raw material (i.e. in a mixed state) is also introduced into the heating wiring 62, nothing is wrong. The heating wiring 62 is held at a temperature in the order of 30 to 40°C by the heater 64b. Also, a pressure in the heating wiring 62 is held at 100 to 300 atm. Under such temperature and pressure conditions, the liquid carbon dioxide is changed to a supercritical fluid in a short time. The liquid raw material passes through the spiral heating wiring 62 at a speed in the order of one minute to several minutes. Through the change of the liquid carbon dioxide dissolved in the liquid-form raw material into the supercritical fluid, a portion of protein as an active substance of the enzymes contained in the liquid-form raw material is destructed to thereby allow a portion of the microorganisms to die out. However, at this time point, the effect is limited to a portion.

[0049]

The liquid-form raw material flowed out from the liquid outlet 54d of the process unit 54 passes through the product collecting path 64 and flows into the decompression tank 65 through the pressure controlling valve 63. At this time, since

the pressure is suddenly lowered, the supercritical state is released and the carbon dioxide is vaporized at a stroke to thereby suddenly expand cubically. At that time, the protein of the enzymes is destructed to thereby cause the microorganisms to die out. Thus, the deactivations of the enzymes and spores and sterilization of the microorganisms can be carried out. Also, when the decompression is carried out, the temperature of the liquid-form raw material is suddenly lowered to a room temperature or lower. The processed product thus obtained is taken out from the product outlet 65c and collected into the product tank 66. The product stored in the product tank 66 has an extremely low rate of active enzymes and no undesirable microorganisms. Also, since no heat is applied when the carbon dioxide is vaporized at the decompression tank 65, the flavor component itself contained in the liquid-form raw material scarcely volatilizes, so that the flavor is not spoiled or lost.

[0050]

Incidentally, as described above, since the carbon dioxide dissolved in the liquid-form raw material is vaporized by the decompression and almost all the carbon dioxide is separated from the liquid-form raw material, the product taken out of the product outlet 65c contains an extremely small amount of the carbon dioxide. However, depending on a kind of the product, it is further required to completely remove the remaining carbon dioxide. In order to produce this type of product, a unit for subjecting the product taken out of the product outlet 65c to a deaeration process may be added at a later step than the decompression tank

65. Such a deaeration unit can be structured by using a conventionally known deaeration method of a liquid, such as a decompressing method (a closed tank in which a liquid is sealed up is evacuated to thereby take out gas from the liquid); or a centrifugation method.

[0051]

The carbon dioxide vaporized in the decompression tank 65 as described above flows into the carbon dioxide collecting path 68 through the carbon dioxide discharge port 65d and is guided to the recycling tank 69 through the pressure controlling valve 67. After a sufficient quantity of carbon dioxide is collected in the recycling tank 69, when the valve 72 disposed on the recycling pipe 70 is opened, the carbon dioxide stored in the recycling tank 69 is again fed to the raw material supply pipe 53 through the recycling pipe 70 and the carbon dioxide supply pipe 60 by the action of the pump 52. At this time, even if carbon dioxide is in a gaseous state in the recycling pipe 70, the carbon dioxide is liquefied when it passes through the cooler 58 and supplied to the raw material supply pipe 53. As described above, the recycling tank 69 can be used as a second liquid carbon dioxide supply source. Therefore, for example, after a sufficient quantity of carbon dioxide is stored in the recycling tank 69, the carbon dioxide stored in the recycling tank 69 is mainly used and only an insufficient quantity is taken out from the bomb 55 to thereby reduce a consumption of the liquid carbon dioxide.

[0052]

In the apparatus as shown in Fig. 3, although solution of the liquid carbon dioxide into the liquid-form raw material is accelerated by allowing the liquid carbon dioxide to be fine bubbles through the filter 61 disposed in the raw material supply pipe 53, there may be employed other acceleration methods for dissolving the liquid carbon dioxide. Fig. 6 is a block diagram showing an example of a solution acceleration mechanism formed of a stationary-type mixer. In the mechanism, the stationary-type mixer 75 is located on the way of the raw material supply pipe 53 on the downstream side than a connection portion of the raw material supply pipe 53 and the carbon dioxide supply pipe 60. Incidentally, in case the stationary-type mixer as described above is used, in order to raise a solution efficiency of the liquid carbon dioxide, for example, a plurality of liquid carbon dioxide supplying pipes formed by diverging the carbon dioxide supply pipe 60 on the downstream side than the pump 59 is connected to the raw material supply pipe 53, so that the liquid carbon dioxide is supplied to the raw material through the plural liquid carbon dioxide supplying portions.

[0053]

Fig. 7 is a block diagram showing an example of a continuous sterilization process apparatus including a plurality of process units. The apparatus shown in Fig. 7 includes three process units 81A, 81B, 81C, each of which has the same structure as that of the process unit 54 used in the apparatus shown in Fig. 3. The raw material supply pipe 53 is diverged into three branch pipes 82A, 82B, 82C on the downstream side than the filter 61. Forward ends

of the three branch pipes 82A, 82B, 82C are connected to liquid inlets of the three process units 81A, 81B, 81C, respectively. Also, the three branch pipes 82A, 82B, 82C are provided with valves 83A, 83B, 83C, respectively. On the other hand, liquid outlets of the three process units 81A, 81B, 81C are connected to branch pipes 84A, 84B, 84C for collecting products, respectively. The three branch pipes 84A, 84B, 84C are merged into one product collecting path 84 on the downstream side. The two branch pipes 84B, 84C are provided with three-way valves 85B, 85C. A connecting port of one of the first three-way valve 85B is connected to the branch pipe 82A through a bypass pipe 86B. In the same way, a connecting port of one of the second three-way valve 85C is connected to the branch pipe 82B through a bypass pipe 86C. The respective three-way valves 85B, 85C can switch their flow paths in two directions as shown by arrows A1 and A2. Operations of the above valves 83A, 83B, 83C and the three-way valves 85B, 85C and operations of the respective process units 81A, 81B, 81C (such as heating operation of heaters provided to the respective process units) are controlled by a control device, not shown. The control device includes input devices for inputting information regarding the process, such as a kind and quantity of the liquid-form raw material to be processed; a kind of the process (such as sterilization, deactivation, deodorization and the like); and in case the sterilization process is carried out, kind of a microorganism to be killed or the like so that a user can input.

[0054]

In the apparatus shown in Fig. 7, the user can properly change the number of the process units to be used and a flow path structure based on the information regarding the process inputted to the controlling device through the inputting devices. For example, in case a quantity of the liquid-form raw material to be processed is large, all three valves 83A, 83B, 83C are opened, and at the same time the directions of the three-way valves 85B, 85C are set to A1. When the flow paths are structured as described above, since the three process units 81A, 81B, 81C are connected in parallel, a large quantity of the liquid-form raw material can be processed at the same time. Also, in case microorganisms to be killed are very strong, the first and second valves 83A, 83B are closed, the third valve 83C is opened, and directions of the three-way valves 85B, 85C are set to A2, respectively. Thus, since the three process units 81A, 81B, 81C become a state where they are connected in series, the processing time becomes long to thereby positively kill even the strong microorganisms.

[0055]

Incidentally, in the explanation for the apparatus shown in Fig. 3, it is mentioned that the time (process time) taken when the liquid-form raw material passes through the process unit 54 is from one minute to several minutes. However, in case plural process units are provided as in the apparatus shown in Fig. 7, since it is enough that a sufficient processing time is taken in the whole process units, the processing times taken by the respective process units can be shortened than the above-mentioned processing time. Therefore, in case an apparatus using a

plurality of process units is structured, the process units having small process tanks may be used instead of the process units having the spiral heating wirings.

[0056]

As described above, when the critical process portion is formed of a plurality of process units, a different temperature can be set for every process unit. Taking advantage of this, for example, when a process unit is shifted to the next process unit, a temperature is suddenly changed, so that bacteria having a lower tolerance with respect to sudden temperature change are shocked to thereby deactivate. Also, a different pressure may be set for every process unit.

[0057]

Incidentally, in the apparatus shown in Fig. 7, the critical process portion is structured by using a plurality of the same process units as that of the apparatus shown in Fig. 3. In the same manner, in the apparatus shown in Fig. 1, the critical process portion may be formed of a plurality of the process units.

Also, the dissolving portion may be structured by a plurality of independently operable units (solution units).

[0058]

The other examples of the apparatus according to the present invention, wherein a part thereof is structured by a plurality of the independently operable units, are shown in Figs. 8 and 9. Incidentally, Figs. 8 and 9 show only an essential part including the solution portion, critical process portion and decompression

portion of the apparatus according to the present invention, respectively.

[0059]

In the apparatus shown in Fig. 8, two sets of solution/process units 93A, 93B including a solution unit 91 and a process unit 92, respectively, are connected in parallel to the raw material supply pipe and the product collecting pipe. In the apparatus, either one or both of the solution/process units can be selected as an operation unit or operation units by properly closing or opening valves 94 according to purposes of the process.

[0060]

In the apparatus shown in Fig. 9, both solution portion 96 and critical process portion 97 are formed of a plurality of units. The solution portion 96 includes two solution units 96A, 96B, a bypass pipe 96C, two valves 96D, 96E, a three-way valve 96F and the like. In the solution portion 96, the valves 96D and 96E are properly opened or closed according to the object of the process and a direction of the three-way valve 96F is suitably changed, so that only either one of the solution units can be selected as an operation unit, or two solution units can be connected in parallel or in series. The critical process portion 97 is also formed of two process units and the like as in the solution portion 96, and a flow path structure can also be changed as in the solution portion 96.

[0061] Incidentally, it is a matter of course that various other unit structures are considered in addition to the structures as shown in Fig. 8 and Fig. 9.

[0062]

There will be explained a structure wherein an acting time of the liquid carbon dioxide can be extended with respect to a substance (enzymes, bacteria or the like) to be processed which are contained in the liquid-form raw material. For example, in order to extend the acting time of the liquid carbon dioxide in the apparatus shown in Fig. 1, a baffle may be provided in the solution tank 11, or another tank (holding tank) for holding therein the liquid-form raw material in which the liquid carbon dioxide has been dissolved for a predetermined time, may be disposed between the solution tank 11 and the heating wiring 20. As described above, in case the acting time of the liquid carbon dioxide is extended, since an efficiency of the critical process is raised, a structure of the critical process portion can be simplified. Therefore, instead of the spiral heating wiring used in the above respective embodiments, for example, a tank can be used to constitute the critical process portion at a low cost.

[0063]

Fig. 10 is a diagram showing an example of a continuous process apparatus including a solution tank, a holding tank and a process tank. In the apparatus shown in Fig. 10, a solution tank 101 has substantially the same structure as the solution tank 11 of Fig. 1. A holding tank 102 includes an introduction port 102a and a carbon dioxide discharge port 102b on an upper portion and a liquid outlet 102c on a lower portion thereof. The introduction port 102a of the holding tank 102 is connected to a liquid outlet 101a of the solution tank 101 through a pipe 103. Outlets of

branch pipes 104 from a carbon dioxide supply pipe, not shown, are located to bottom portions of the solution tank 101 and holding tank 102, respectively, and mesh-type filters (which may be the same filter as that used in the apparatus shown in Fig. 1 or 3) 105 are provided to the outlets thereof, respectively. The process tank 106 includes an introduction port 106a at the bottom portion thereof and a liquid outlet 106b at the upper portion thereof. The process tank 106 is provided with a heater 107 for heating an interior thereof. A liquid outlet 106b of the process tank 106 is connected to a decompression tank (which may be the same as that used in the apparatus shown in Fig. 1 or 3) 109 through a product collecting pipe 108. Although a mechanism for feeding the liquid-form raw material to the solution tank 101, a mechanism for feeding the liquid carbon dioxide to the solution tank 101 and the process tank 106 and a mechanism for collecting and recycling the carbon dioxide discharged from the respective tanks are not shown, these mechanisms may be structured, for example, in the same manner as shown in Fig. 1.

[0064]

In the apparatus as shown in Fig. 10, while the liquid-form raw material is continuously supplied to the solution tank 101 through a raw material supply path 100, fine bubbles of the liquid carbon dioxide and the liquid-form raw material are subjected to a parallel flow contact in the solution tank 101 to thereby allow the liquid carbon dioxide to dissolve in the liquid-form raw material. A theoretical value of a time for which the liquid-form raw material (constitutive molecules thereof) pass through the

solution tank 101 can be obtained from a capacity of the solution tank 101 and a supplying flow amount of the liquid-form raw material, or the like. However, practically, when the fine bubbles of the liquid carbon dioxide are elevated in the liquid-form raw material by a buoyancy, a part of the liquid-form raw material goes with the fine bubbles so that it passes through the solution tank 101 quicker than the other liquid-form raw material portion. Thus, the time for which the liquid-form raw material passes through the solution tank 101 is varied due to supply of the liquid carbon dioxide. According to setting of supply quantities of the liquid-form raw material and the liquid carbon dioxide, the dispersion of the passing time of the liquid-form raw material, as described above, may cause dispersions and lowering of a sterilization effect and an enzyme deactivation effect, which may result in a poor quality of products.

[0065]

Therefore, in the apparatus as shown in Fig. 10, the liquid-form raw material taken out from the solution tank 101 is held in the holding tank 102 for a fixed time to thereby allow the liquid carbon dioxide to sufficiently act on the enzymes and bacteria in the liquid-form raw material (to allow the liquid carbon dioxide to sufficiently permeate into proteins constituting the enzymes and bacteria). Thus, even if the liquid-form raw material passes through the solution tank 101 for a time shorter than the theoretical value, since the solution shortage of the liquid carbon dioxide in the solution tank 101 can be sufficiently made up, the sterilization effect and the enzyme deactivation effect in

the critical process step and decompression step can be sufficiently obtained. Further, in the apparatus shown in Fig. 10, the liquid-form raw material is subjected to a countercurrent contact with the fine bubbles of the liquid carbon dioxide in the holding tank 102. In this method, according to flow of the liquid carbon dioxide (fine bubbles thereof), the liquid-form raw material is properly stirred to thereby allow the liquid carbon dioxide to be uniformly dissolved in the whole liquid-form raw material. Incidentally, it is not essential that the liquid-form raw material is subjected to the countercurrent contact with the fine bubbles of the liquid carbon dioxide in the holding tank 102. A good effect can be obtained even when the liquid-form raw material is merely held in the holding tank 102 for a fixed time.

[0066]

Hereinabove, although embodiments of the present invention have been explained, the present invention is not limited to the above embodiments. For example, in the solution acceleration mechanism shown in Fig. 6, although the stationary-type mixer is used, it is also possible to structure the solution acceleration mechanism by a mixer with a stirring member. Also, instead of the filter, for example, the fine bubbles of the liquid carbon dioxide may be generated by using an ultrasonic generating apparatus. Further, in the above embodiments, although the heating wiring (20, 62) is shaped in a spiral form, the heating wiring is not limited thereto and may be formed in other shapes.

[Brief Description of the Drawings]

[Fig. 1] is a diagram showing a structure of a continuous enzyme deactivation process apparatus;

[Fig. 2] is a table showing results obtained by testing ratios of active enzymes remaining in the liquid-form raw material processed by the apparatus as shown in Fig. 1;

[Fig. 3] is a diagram showing a structure of another continuous enzyme deactivation process apparatus;

[Fig. 4] is an enlarged view of a portion (a portion enclosed by a rectangle R shown in Fig. 3) wherein a filter is disposed in a raw material supply pipe;

[Figs. 5] (a), (b) are diagrams showing a plurality of filters disposed in a raw material supply pipe, respectively;

[Fig. 6] is a diagram showing an example of a solution acceleration mechanism structured by using a stationary-type mixer;

[Fig. 7] is a block diagram showing an example of a continuous sterilization process apparatus including a plurality of process units;

[Fig. 8] is a diagram showing another continuous process apparatus, a part of which is structured by a plurality of units;

[Fig. 9] is a diagram showing another continuous process apparatus, a part of which is structured by a plurality of units; and

[Fig. 10] is a diagram showing an example of a continuous process apparatus including a solution tank, a holding tank and a process tank.

[Explanation of Symbols]

1, 51 ... raw material tank
3, 53, 100 ... raw material supply path (raw material supply pipe)
10, 60 ... carbon dioxide supply path (carbon dioxide supply pipe)
11, 101 ... solution tank
13, 61 ... filter
19, 54b, 107 ... heater
20, 62 ... heating wire
22, 63 ... pressure controlling valve
23, 64, 84 ... product collecting path
24, 65 ... decompression tank
26, 66, 109 ... product tank
27, 68 ... carbon dioxide collecting path
28, 67 ... pressure controlling valve
29, 69 ... recycling tank
102 ... holding tank
106 ... process tank

[Name of Document] Statement of Abstract

[Abstract]

[Object] In a continuous process method and apparatus of a liquid-form raw material using a liquid carbon dioxide in a supercritical or subcritical state, high sterilization and enzyme deactivation effects can be obtained even with a small process tank.

[Solving Means] There are separately provided a solution tank (101) for dissolving a liquid carbon dioxide into a continuously supplied liquid-form raw material; a solution tank (101) for holding, for a predetermined time, the liquid-form raw material into which the liquid carbon dioxide has been dissolved to thereby allow the liquid carbon dioxide to penetrate into enzymes, microorganisms or the like in the liquid-form raw material; and a process tank (106) for holding the liquid-form raw material from the holding tank (102) under predetermined temperature and pressure conditions to thereby transform the carbon dioxide into a supercritical or subcritical state.

[Selected drawing] Fig. 10

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Yutaka Osajima et al.
Title : CONTINUOUS PROCESSING METHOD AND
CONTINUOUS PROCESSING APPARATUS FOR
: LIQUID-FORM SUBSTANCE, AND LIQUID-FORM
SUBSTANCE PROCESSED THEREBY
Serial No. : 09/684,433
Filed : October 10, 2000
Group Art Unit : 1744
Examiner : Elizabeth L. McKane

VERIFICATION OF TRANSLATION

Sir:

I, Teruko Hashimoto, residing at 1900 South Eads Street, Arlington, Virginia 22202, U.S.A., declare that I am fluent in Japanese and English, and that a herewith submitted English translation of Priority Document No. 2000-127082 filed on April 27, 2000 for the above-identified patent application is a true and accurate literal translation.


Teruko Hashimoto

Date: September 9, 2004